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REFRACTIVE INDEX DETERMINATION BY ORIENTATION VARIATION. 1. UNIAXIAL CRYSTALS

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ABSTRACT

A transparent, uniaxial crystal is ground to a small, almost perfect sphere. It is diametrally measured and immersed in a medium which has a refractive index that lies somewhere between the principal indices of the crystal. The sphere is optically oriented on a five-axes universal stage and rotated in monochromatic light until a non-principal refractive index of the crystal equals the index of the medium. The angle of rotation between the non-principal and a principal index of refraction is recorded and the path-difference from this known orientation and from the principal section are measured with a variant compensator. These path-differences, the angle of rotation, the nonprincipal index of refraction, and the diameter of the sphere are used on nomograms; and all critical optical parameters may then be rapidly and accurately derived. The principal birefringence may be determined independently, before any refractive index is ascertained. Refractive indices may be determined in less than an hour with an accuracy better than ± 0.0005.

BASIS OF METHOD

Single variation as applied to the determination of refractive indices under the polarizing microscope was first proposed by Merwin (1922) with later application by Eskola (1922), Tsuboi (1923), and others. In this method wave-length of monochromatic light is varied progressively until a condition is reached in which an immersed crystal and the immersion medium have the same refractive index. Later Emmons (1926) achieved the same end by varying the temperature of the immersion medium. A third type of approach to single variation, set forth in this paper, is progressive change of the optical orientation of the crystal until an intermediate, nonprincipal refractive index of the crystal is equivalent to the index of the medium in which it is immersed.

Most of the principles used in single variation by orientation are similar to those used in various universal stage techniques described by Emmons (1943). The large departure in this paper is that the crystal grains to be studied by orientation variation must be ground into essentially perfect spheres before mounting them on the universal stage. Such sphericity assures a known, constant thickness of section for any conceivable optical orientation, thus permitting conversion of orientation variation data to absolute values of refractive indices.

Orientation variation, in essence, permits the microscopist to indirectly deduce ϵ and ω, the principal refractive indices of an unknown uniaxial crystal[1]—in other words to find the lengths of the semimajor and semi-

[1] This paper is limited to uniaxial crystals. Modification of equipment is being made in order to overcome certain problems imposed by biaxial crystals.

minor axes of the indicatrix. This is done with the aid of nomograms from three components the measurements of which are determined with the polarizing microscope, 5-axis universal stage, and various accessories. These components are: 1) the length of an intermediate central-radius of the indicatrix ϵ', 2) the angle of arc between this central-radius and the semimajor or semiminor axis of the indicatrix ϕ, and 3) the difference between the length of this central-radius and one of these two critical semiaxes ϵ'-ω.

Continuing with the three components in the order enumerated: 1) the length of the central-radius is measured in the principal section of the indicatrix and is determined by progressively manipulating the universal stage until a non-principal refractive index, ϵ', of the spherical grain matches the known refractive index of the medium in which it is immersed; 2) the angle of arc, ϕ, between this central-radius, ϵ', and a critical semiaxis, ω, is read from a Wright arc or from the graduated drum of the universal stage; and 3) the difference between the lengths of the intermediate central-radius and the semimajor or semiminor axis of the indicatrix is determined by measuring the diameter of the sphere with a measuring ocular and ascertaining the amount of path-difference between ϵ' and ω with a variant compensator. This parameter is a function of thickness of section and path-difference,

$$(\epsilon' - \omega) = \frac{\text{path difference between } \epsilon' \text{ and } \omega}{\text{diameter of the sphere}}$$

Component 3, the length difference between the intermediate central-radius and a critical semiaxis of the indicatrix, $(\epsilon'$-$\omega)$, is algebraically added to the intermediate central-radius, thus yielding one of the critical semiaxes of the indicatrix, ω.

By means of the equation of the ellipse,

$$\epsilon' = \frac{\epsilon\omega}{\sqrt{\omega^2 \sin^2 \phi + \epsilon^2 \cos^2 \phi}},$$

the second critical semiaxis, ϵ, of the uniaxial indicatrix may be found. To simplify this calculation, Emmons (1943, Pl. 10) has provided an invaluable nomogram. Unfortuantely, the higher the birefringence of the crystal sphere and the greater the extrapolation, the less the accuracy of the value of the second critical semiaxis, ϵ. Consequently, it is often advantageous to measure the difference in length of the critical semiaxes—in other words principal birefringence of the sphere,

$$(\epsilon - \omega) = \frac{\text{path difference between } \epsilon \text{ and } \omega}{\text{diameter of the sphere}},$$

—and algebraically add this value to the first critical semiaxis, ω, in order to determine a more accurate value for the second semiaxis, ϵ.

Sample Preparation

Fragments of the study specimen are to be crushed by pounding them in an agate mortar. If the supply of specimen is quite small, the fragments may be ground in a mortar containing an appropriate liquid, and although grinding rather than pounding the fragments is not desirable, it does prevent any significant loss of the sample during the crushing operation. The crushed fragments are then sieved through a nest of 3-in Tyler sieves and the 0.3 to 0.7 mm fraction is retained for rounding. A load not to exceed four to six grains of the crushed fragments is placed in an air-driven sphere grinder (to be described later), and abraded. The resulting spheres usually range in diameter from 0.1 to 0.3 mm. If the load of grains is rounded in the grinder for an unusually long period of time, perhaps eight or ten minutes, excellent spheres 0.03 to 0.02 mm in diameter, or smaller, may be obtained.

If the variant compensator used in the orientation control procedure is limited to three or four orders, it is obviously necessary to grind crystals with very high birefringence into exceedingly small spheres.

While the crushed grains are rounding and polishing in the sphere grinder, several fragments of the specimen may be taken from one of the sieve fractions and examined under the polarizing microscope. This cursory examination allows the microscopist to establish a crude estimate of the optical parameters of the unknown crystal, and thus to select the immersion medium to be used for mounting the finished spheres on the universal stage.

The selected immersion medium should have a refractive index that lies approximately midway between the limiting refractive indices of the study specimen. Such a medium will cause the value of ϕ—the angle between ϵ' and ω—to be at least 30°. Consequently, if the microscopist elects or is forced to determine ϵ via extrapolation, the value of ϵ derived by this extrapolation will be reasonably accurate.

The most nearly perfect of the completed spheres are selected under a binocular microscope, then are hand-picked and transferred from the grinder to a small glass vial with the aid of a moistened camel's hair brush. The spheres frequently acquire a significant static electrical charge and extreme care must be taken or they may be repelled and lost.

Mounting and Measuring the Spheres

Two or three spheres of different sizes are mounted on the universal stage. A very thin camel's hair brush, slightly moistened by barely touching the surface of the chosen immersion medium, is used to transfer the spheres to a thin glass slide[1] which serves as a protective lower cover glass and rests with immersion fluid contact on the inner glass plate of the universal stage. The slide should be shortened so that it is approximately 55 to 60 mm long, which is 10 to 15 mm longer than the slide commonly used for universal stage work. This extra length causes the slide to protrude from beneath the mount, this protrusion serving as a handle to expedite rolling and centering the spheres while they are mounted between the hemispheres of the universal stage.

The diameter of each sphere is measured to an accuracy of at least 0.001 mm by using an image-splitting measuring eyepiece which is inserted into the polarizing microscope tube in place of the standard ocular. This special eyepiece will be discussed later.

It is imperative that the spheres be measured "dry". They must not be measured covered with immersion fluid or when mounted between the hemispheres of the universal stage because this will cause magnification and induce a serious error.

[1] Corning No. 2950 glass slides are suitable for this purpose.

After the "dry" diameter of each sphere has been recorded, the mount may be completed. The immersion medium and the upper cover glass are placed on the spheres, more medium is placed on the top of the cover glass, and the upper hemisphere of the universal stage is added. The sphere to be studied must be centered under the intersection of the microscope's cross lines. A gentle manipulation of the thin glass slide greatly facilitates this important step. It is absolutely essential that the sphere be exactly centered throughout the entire procedure, and that the microscope and universal stage be in perfect alignment, otherwise the crystal cannot be precisely oriented on the universal stage, and measurement of path-difference cannot be accomplished with sufficient precision.

The largest of the crystal spheres in the mount usually supports most of the weight of the upper hemisphere, and consequently becomes badly strained; in fact, the largest cyrstal sphere is frequently strained to the point that it becomes biaxial. The upper cover slip of the mount warps slightly over this largest sphere, and in the immediate warped area, the slip is strained into anisotropism.[1] This problem can be solved by one of two means. Either thumb screws and the metal housing of the upper hemisphere can be modified so that the weight of the hemisphere is supported by the thumb screws; or the thin glass slide which serves as a lower cover slip can be manipulated until one of the smaller spheres in the mount becomes centered under the cross lines and lightly wedged between the thin glass slide and the upper cover slip.[2]

Procedure

Step 1. The crystal sphere is oriented on the universal stage so that the optic axis is horizontal and north-south, that is, normal to the axis of the microscope and in the plane of the stage. It is advisable to use a half-shadow accessory in conjunction with a Wright universal eyepiece and cap analyzer in this and all other appropriate orientation operations. The microscopist must bear in mind that one of the major sources of error in this single variation procedure, as in any universal stage procedure, may be his inability to determine the precise point of extinction, and consequently, he cannot reach an exact desired orientation. The solution of this vital problem is the use of a Nakamura half-shadow plate or a Máce de Lépinay half-shadow wedge. Thus, near perfect-extinction, under good conditions within 0.05°, and critical orientation, to within 0.1°, are assured.

Step 2. After the crystal sphere is oriented the principal birefringence is determined. The crystal is rotated exactly 45° on the microscope stage into the "compensating" position and the path difference between ϵ and ω is measured with a modified Wright combination quartz wedge. This variant compensator will be considered later. The principal birefringence is determined from the standard equation, $(\epsilon\text{-}\omega)\ \Delta/T$, where $(\epsilon\text{-}\omega)$ is the principal birefringence, Δ the path-difference in nm, and T the thickness of section (diameter of the sphere) in mm. Or the calculation can be made from a nomogram patterned after the Michel-Lévy birefringence chart.

Step 3. The optical orientation is varied until the refractive index of the sphere is equal to that of the immersion fluid. First, the microscope stage is returned from the "compensating" position so that the optic axis is again precisely north-south and horizontal and the refractive index of the crystal is ϵ. From this position a north-south rotation on the outer east-west axis of the universal stage will cause the refractive index of the sphere to vary from ϵ to ω, the limiting indices. A critical point will be reached along this north-south rotation where the refractive index of the crystal, ϵ', exactly matches that of the medium,

[1] Lienhart, D. A. (1964) Unpublished M.S. Thesis, University of Cincinnati.

[2] Perhaps the universal stage spindle described by Wilcox (1959) may provide a better means for holding a perfectly centered sphere securely without inducing strain.

thus giving the refractive index of ϵ'.[1] The angle of rotation on the outer east-west axis from the ϵ to the ϵ' position, the complement of ϕ, is read from the graduated drum of the universal stage and then corrected to the true "crystallographic" angle of rotation. The temperature of the immersion medium in the mount is read and recorded so that the refractive index of the medium can be corrected for the thermal coefficient of refraction.

Step 4. The refractive index of ω is determined by algebraically adding the partial birefringence, $(\epsilon'-\omega)$, to ϵ'. With the axes of the universal stage locked in the position where the refractive index of the sphere, ϵ', matches the index of the fluid, the microscope is rotated exactly 45° to the "compensating" position and the path-difference between ϵ' and ω is measured with the aid of the Wright combination wedge. The partial birefringence, $(\epsilon'-\omega)$, is determined in the usual manner from the equation $(\epsilon'-\omega)=\Delta/T$, or from the appropriate nomogram. The refractive index of ω is determined by algebraically adding this partial birefringence to the refractive index of ϵ'.

Step 3. The index of refraction of ϵ is determined by algebracially adding the principal birefringence to the refractive index of ω, or as previously stated, by extrapolation, that is, solving the equation for the ellipse or by use of the Emmons nomogram (op. cit.).

A typical example using quartz as the study crystal illustrates the recorded data and the calculations for each of the above steps in the orientation variation procedure.

Step 1. Orient the quartz sphere so that the optic axis is horizontal and north-south.

Step. 2. Measure the principal birefringence of the crystal.

Compensation (path-difference) ϵ to $\omega=2224.9$ nm

$$(\epsilon-\omega)\text{ principal birefringence}=\frac{\Delta(\text{path difference})}{T(\text{diameter of sphere})}=\frac{2224.9\text{ nm}}{0.2445\text{ mm}}=0.0091$$

Step 3. Determine ϵ' and ϕ.

ϕ corrected (degrees from ω to ϵ') = 33.7°

Temperature of mount = 18.7°C

ϵ' corrected for temperature = 1.5469 at 20°C

Step 4. Determine the refractive index of ω.

Compensation (path-difference) ϵ' to $\omega=639.9$ nm

$$(\epsilon'-\omega)\text{ partial birefringence}=\frac{\Delta(\text{path difference})}{T(\text{diameter of sphere})}=\frac{639.9\text{ nm}}{0.2445\text{ mm}}=0.0026$$

$$\omega=\epsilon'\pm(\epsilon'-\omega)=1.5469-0.0026=1.5443$$

(Quartz is a positive mineral and $(\epsilon'-\omega)$ must be subtracted from ϵ'.
If the crystal under study had a negative sign, $(\epsilon'-\omega)$ would be added to ϵ'.)

Step 5. Determine ϵ. Preferred solution:

$$\epsilon=\omega\pm(\epsilon-\omega)=1.5443+0.0091=1.5534$$

Alternate solution by extrapolation:

$(\epsilon-\omega)$ via solving the ellipse equation or by Emmons' nomogram = 0.0090

ϵ determined from principal birefringence extrapolation = 1.5533

Notice that the principal birefringence determined by extrapolation and the value of ϵ based upon this extrapolation differ by 0.0001 from the more accurate and therefore preferred solution.

[1] The Becke line method (central illumination) is used to determine the refractive index match. Several oblique illumination techniques were tried, and these, though useful, proved to be not as accurate as central illumination.

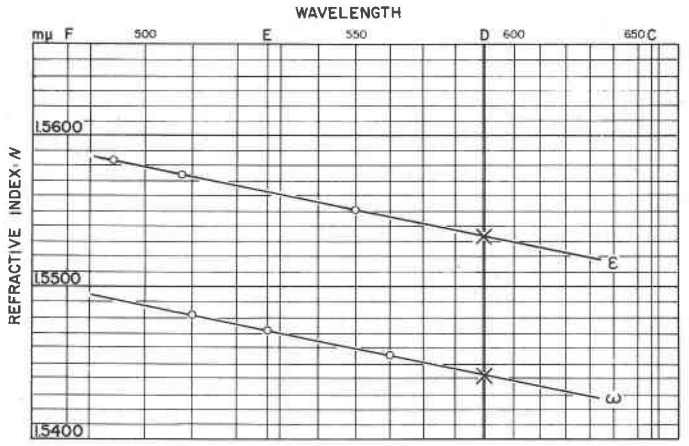

FIG. 1. Hartman net graph of quartz standard presented in the five step summary. This illustrates the agreement of orientation variation data and double variation data (X = orientation variation data and O = double variation data).

Dispersion of the principal refractive indices is a discriminating optical parameter. Curves for this important optical property may be derived by changing the wave-length of the monochromatic light source and repeating the orientation variation procedure just outlined. The compensator and immersion media, of course, must be calibrated for any new wave-length of monochromatic light used.

Figure 1 derived from determining a quartz standard by orientation variation and by double variation attests the accuracy of the orientation variation technique.

In order to achieve the desired accuracy for refractive indices, the temperature of the immersion medium in the mount must be known to better than 0.5°C. This poses a problem which can be solved by using media characterized by low thermal coefficient of refraction, or by finding a convenient method to measure with accuracy the temperature of the high-coefficient, double-variation media currently used. Various arrangements and types of temperature-measuring equipment, including a millivolt potentiometer equipped with a copper/constantan thermocouple, have been used to solve this problem. Although the results from the potentiometer have been excellent, so much time is consumed in the constant calibration of the reference position of the potentiometer[1] that a more efficient method is needed. Empirical laboratory observation has convinced the writer that reasonably equivalent results can be had by adding a 1.5°C factor to the temperature read from a small, accurate, mercury therometer taped to the arm of the microscope; however, this thermal correction factor might not be valid for a different optical set up or a different laboratory situation.

MODIFIED WRIGHT COMBINATION QUARTZ WEDGE

The zero- to four-order modified Wright combination quartz wedge[2] consists of a zero- to five-order wedge shaped plate of quartz mounted over a one-order quartz plate of uniform thickness. The fast-slow directions of the two plates are opposed so that this combina-

[1] Wilson, D. A. (1967) Unpublished M.S. Thesis, University of Cincinnati.

[2] Available from E. Leitz.

tion quartz wedge has a sharp line at the zero position rather than the inevitable jagged, irregular broken thin edge universally found in standard quartz wedges. The two quartz plates are mounted in "club sandwich" fashion between three glass cover slips. Two of the covers have the shape of a counter wedge so as to eliminate image displacement caused by the usual shallow prism shape of standard quartz wedges. The modified combination wedge is especially designed to be used in the accessory slot of a Wright ocular. Here the photo-engraved divisions of the scale on the top cover slip may be accurately read under magnification in the focal plane of the eyepiece.

Early attempts to do orientation control were not entirely successful, one of the primary reasons being that path-difference could not be accurately measured; consequently, numerous research-model variant compensators of both the wedge and Nikitin tilting type were tested in monochromatic light by checking the value of K (constant for the compensator) every quarter wave length ($\lambda/4$) throughout the excursion of the instrument. The linkage of the tilting compensators, in general, proved to be so poor that successive values of K deviated as much as ± 32 nm, even in some of the better instruments.[1] Wedge variants were found to be more accurate for two reasons: they have no linkage; and with progressive insertion for compensation the retardation of the wedge increases as a linear rather than ellipsoidal function, as in a tilting variant. A modified Wright combination wedge was repeatedly checked at successive $\lambda/4$ increments and was found to have a maximum deviation in K of only ± 3 nm.[2]

Standard research-model variant compensators have an upper compensation limit of four orders or less. Though high-order, thick-plate, tilting compensators and high-order obtuse wedges can be purchased from most manufacturers, nevertheless, it is axiomatic that the higher the maximum order of the variant compensator the less the accuracy of the instrument. Because of the four-order limit of the accurate modified Wright combination wedge only minerals with low to medium birefringence, and only small diameter, spheres can ordinarily be studied by orientation variation. To solve this problem and increase the range of the Wright wedge, four- and eight-order quartz plate compensators[3] of known path-difference may be inserted below the Wright wedge, in the accessory slot of the microscope tube, sub-stage assembly, or objective. This increases the thickness of section without affecting the gentle slope and acute angle of the wedge and extends the upper compensation limit to eight-, twelve- or sixteen-orders, without seriously, imparing accuracy.

Image-Splitting Measuring Eyepiece

The design of an image-splitting measuring eyepiece is similar to that of a common split-image range finder in a camera. This ocular is essentially a prism assembly mounted in a conventional compound microscope system which is linked to a micrometer screw and vernier. When the micrometer screw is turned, the prisms are rotated and the two images of a single object in the field of view may be made to transgress one another edge-to-edge. The edge-to-edge interval traveled is the measured distance across the object and may be read from the vernier of the micrometer screw.

The image-splitting measuring eyepiece removes the basic parallax problems common to measuring graticules and filar-type measuring oculars. Thus, the uncertainty of the precise location of the reference line which is just touching the very edge of the object to be

[1] Research-model tilting compensators of all major manufacturers, except the new Leitz magnesium fluoride compensator, were tested.

[2] Swisher, M. M. (1966) Unpublished B.S. Thesis, University of Cincinnati.

[3] Available from E. Leitz at a moderate cost.

measured is eliminated. The problem of instrument rigidity is also removed. Even if the eyepiece is agitated and shifted in the tube of the microscope, the relation between the double images remains fixed.

The image-splitting ocular is said to have an accuracy as high as 0.125 μm, depending upon the numerical aperture of the microscope objective used.[1]

The eyepiece must be calibrated with the aid of a stage micrometer for the various universal-stage objectives of the polarizing microscope.

Air-Driven Sphere Grinder

The air-driven sphere grinder (Figs. 2 and 3) is a small cylindrical chamber lined with abrasive-coated paper, the chamber containing a screened exhaust opening and a minute aperture through which a jet of compressed air enters and forces sand-sized particles to

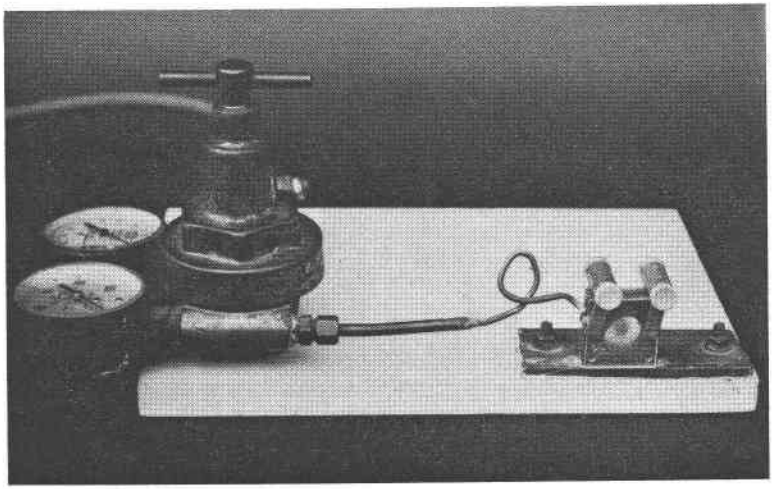

Fig. 2. Air-driven sphere grinder coupled to gas-cylinder pressure regulator. (Photograph by R. J. Starmer)

tumble against the cylinder walls at a high velocity. The grinder was designed by Bond (1951) for use in the Bell Laboratories. In preparing spheres, Bond found that the obvious methods of grinding in grooved laps were impractical. He believed that if the individual particles of the material were set spinning about their centroid by a jet of air, they would spin about their axis of greatest moment of inertia, and thus, when the particles were tossed against the abrasive paper, the parts farthest from the centroid would be abraded fastest, and eventually the material would become spherical. His grinder specifications follow: "A brass plate, $1\frac{1}{2}$ in wide and $\frac{1}{4}$ in thick, has a $\frac{3}{4}$ in hole thorugh it. Air is brought in tangentially, the jet being 0.021 in in diameter. Emery-coated paper, $\frac{1}{4}$ in wide, lines the $\frac{3}{4}$ in hole, one end of the paper being in a slot (to assure that the air does not go under the paper). The other end of the paper almost reaches the jet and is held in place by soft wax between the paper and the brass. Lucite plates cover the faces of the brass plate; one Lucite plate has a $\frac{1}{4}$ in air escape hole covered with a fine silk screen."

In optical work the spheres must have a very fine surface finish; consequently, the

[1] (1963) Vickers Image Splitting Measuring Eyepiece Operating Manual.

medium-grit paper used for initially rounding the spheres in the grinder must be changed to a finer grit paper. Since one of the two Lucite end-plates of the grinder must be removed and replaced to accomplish this end, it is convenient to use helical spring loaded pinch-clamps rather than screws as shown by Bond (Figs. 2 and 3) for holding the Lucite end-plates against the central brass plates of the grinder. It is also advantageous to connect a gas-cylinder pressure regulator between the grinder and the laboratory compressed-air fixture. Gas-cylinder pressure regulators usually have two pressure gauges separated by a precision outlet valve. One pressure gauge is used for measuring the air pressure in the laboratory compressed-air line, and the other gauge and the valve can serve to conveniently and precisely regulate the air pressure in the air-driven grinder. The cylinder pressure regulator and the grinder may be mounted on a single board as shown in Figure 2.

The grinding time, amount of air pressure, and grit size of the abrasive paper necessary for successfully grinding spheres are functions of the physical properties of the specimen,

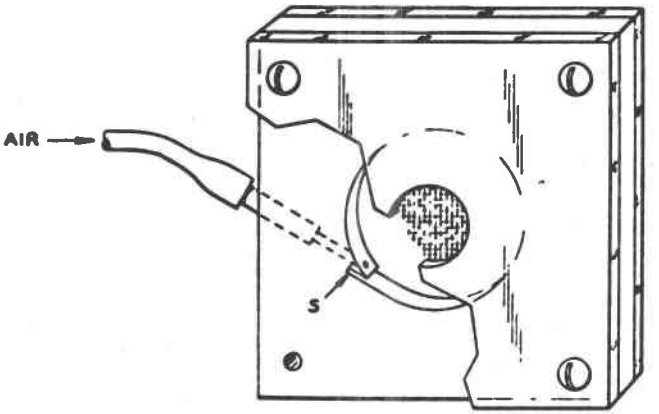

FIG. 3. Air-driven sphere grinder (after Bond).

and are quickly learned through experience. In general, it requires a few seconds to a few minutes grinding time, 50 to 60 lbs per sq in air pressure, and 220 to 400 grit paper to round the spheres. Three or four times this amount of grinding time, reduced air pressure, 25 to 30 lbs per sq in, and extremely fine grit paper or crocus cloth are required for polishing the spheres to a mirror finish.

Spheres thus produced have been very carefully measured with an image-splitting measuring eyepiece and have been found to deviate less than 1 percent from sphericity. This concurs with the findings of Bond (1951).

DISCUSSION

The optical orientation variation method described here is the result of a search for a more rapid analytical technique than the two single variation methods in current use. While orientation variation is not a "panacea," it is rapid, and sufficiently accurate for routine research work. Optical standards have been determined repeatedly by the Emmons' (1929) double variation technique and by orientation variation.

The refractive indices obtained by the two methods were commonly identical (Fig. 1), and most infrequently differed by more than 0.0002, even when determined by lower-division students. Cultured quartz from an exceptionally pure "batch"[1] was used as the uniaxial standard and "home grown" artificial mascagnite $(NH_4)_2SO_4$ as the biaxial. While this paper is not concerned with biaxial crystals, it is appropriate to note that these two standards possess strikingly different physical properties; nevertheless, both can be rapidly and easily ground into almost perfect spheres. Quartz has a hardness of 7 and is characterized by a well-developed conchoidal fracture, whereas mascagnite has one good cleavage [001] and a hardness of only 2. Thus, most minerals, other than mica, kyanite and perhaps a few others, including those with unusual optical properties, can be studied by orientation variation.

The orientation method can be carried to completion, including the derivation of dispersion curves for the refractive indices, in less than an hour. The accuracy of the refractive indices is better than ± 0.0005, and under optimum conditions ± 0.0002.

It is sometimes advantageous to add a third variable—orientation variation—to the Emmons double variation technique, in essence, triple variation. This permits the accurate measurement of the refractive indices of certain high-index crystals which otherwise cannot be readily determined. Questionable optical data can also be conveniently reevaluated by checking the results gained from the use of any one variable against that acquired from the other two. In addition, orientation problems, including those ordinarily caused by cleavage, can be solved rapidly by manipulating the sphere into a more advantageous position in the mount.

Principal birefringence frequently is a discriminating optical parameter. This property has, perhaps, been unduly neglected because of the very real difficulty of preparing critically cut crystal plates, which usually are necessary if this important parameter is measured directly, without first determining limiting refractive indices. Use of crystal spheres obviates this problem. Furthermore, many common crystals too highly refractive to have principal birefringence measured in standard media in the usual manner can be determined easily by orientation variation, that is, by grinding them into spheres and immersing them in any medium that has a refractive index that lies anywhere between the limiting indices of these crystals.

[1] Personal message from Mr. Harry W. Dodds of the Sawyer Research Products Inc. who kindly donated the quartz standard to the University of Cincinnati.

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